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Study of the zeolite A conversion into sodalite under sonohydrothermal conditions

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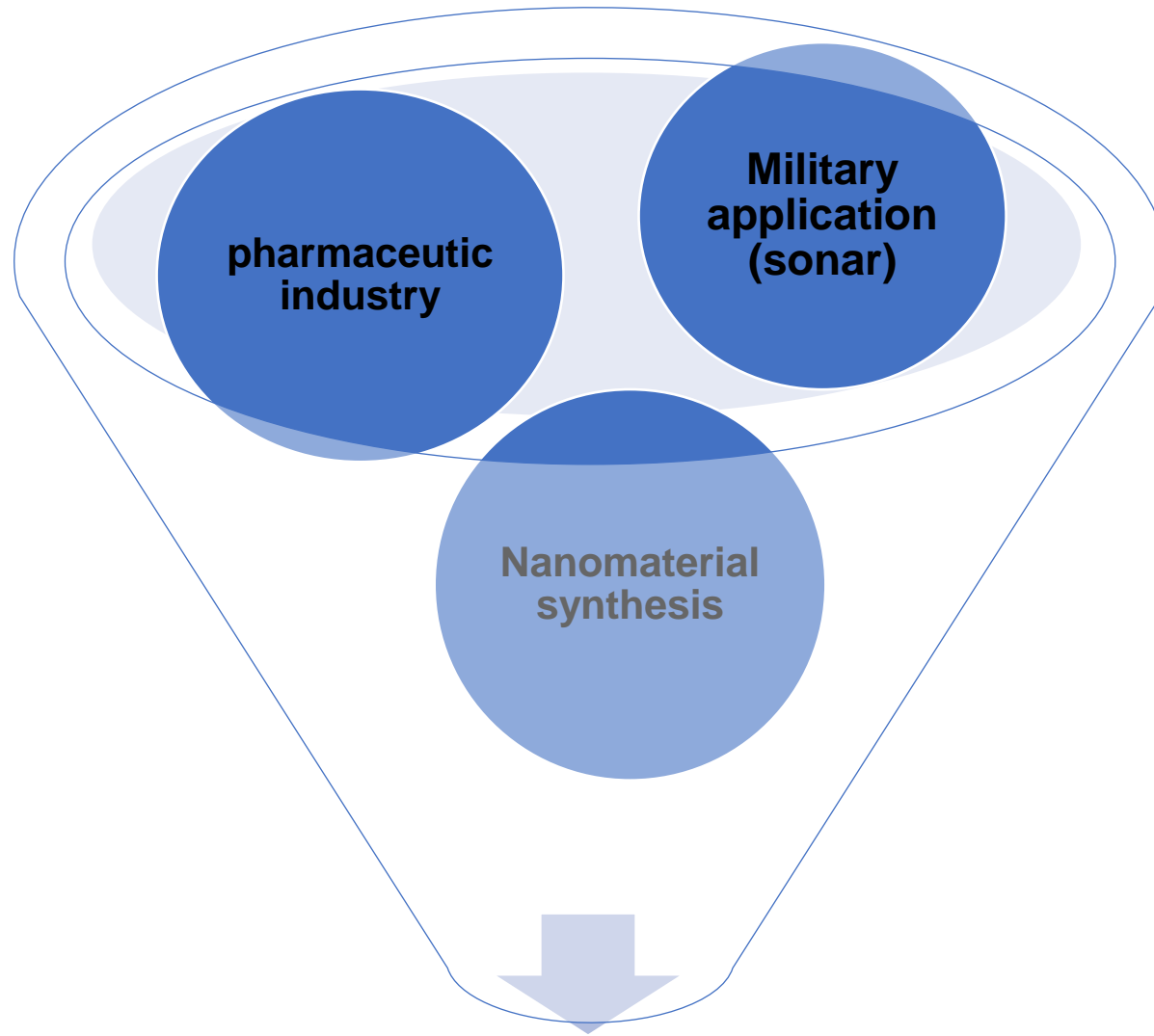
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UNIVERSITÉ DE
MONTPELLIER



IC2MP
Institut de Chimie des Milieux et Matériaux de Poitiers



Ultrasound application

Sonochemistry & Acoustic cavitation



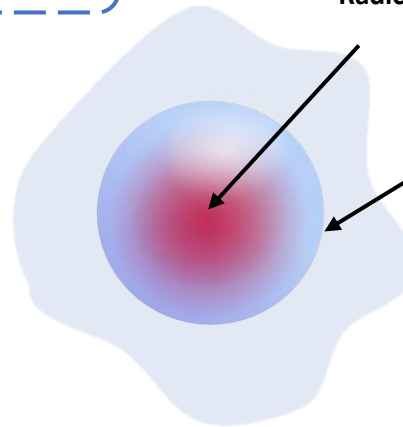
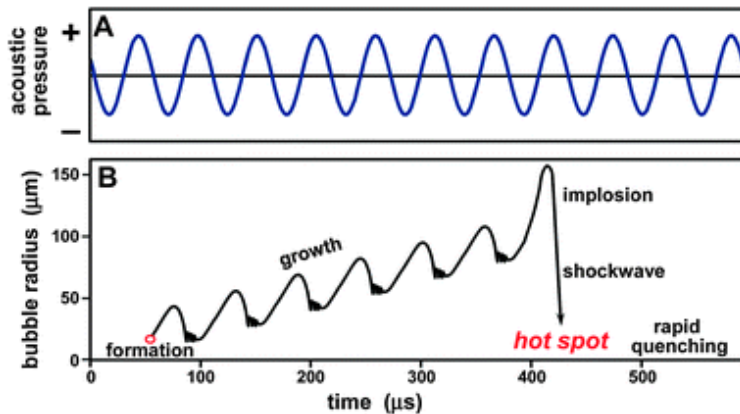
1927: Richards & Loomis describe sonochemical hydrolysis of dimethyl sulphate at **100-500 kHz**

1929: Schmitt describes the ultrasonic oxidation of iodide ions at **750 kHz**

1991: Thèse de Nikonov (Russian Inst. of Physical Chemistry) dedicated to the chemistry of the An under ultrasound

Acoustic Cavitation

Nucleation > growth > Implosive collapse of micro-bubbles



Inside the bubble, gas and steam
Radical formation (e.g. HO^\bullet , H^\bullet , CH_3^\bullet), excited species (e.g. Xe^* , C_2^* , H_2O^*), ionisation...

At the interface
Radical trapping, species thermolysis
non-volatile, hydrothermal reactions

Bulk
Environmental conditions: Secondary reactions

Hot spot Model:

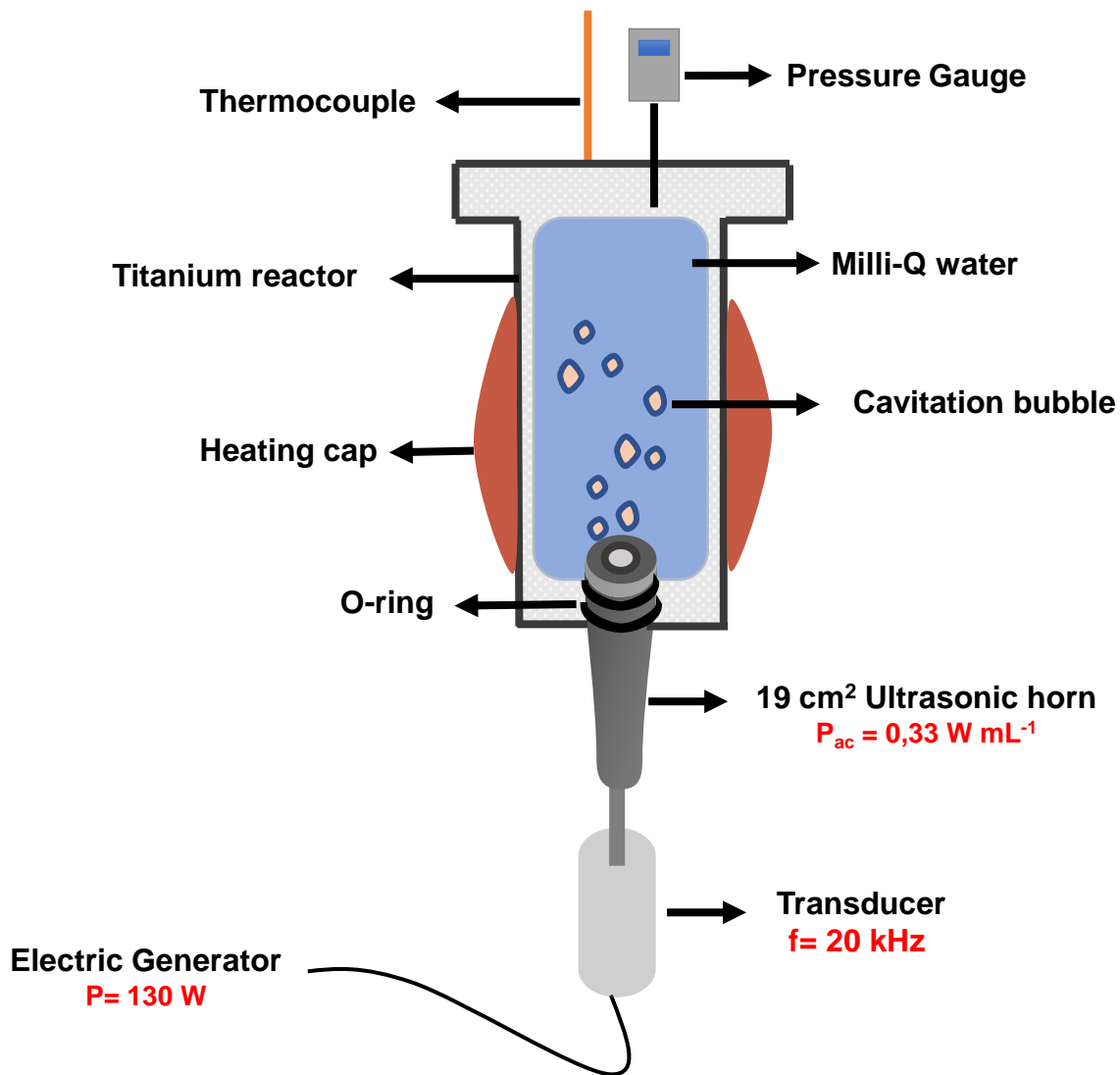
$T \sim 5000 \text{ K}$
 $P \sim 1000 \text{ bars}$

Mechanical force at the interface S/L
→ erosion, fragmentation, depassivation...



Micro-jet: $\sim 150 \text{ m s}^{-1}$ à 20 kHz

Sonohydrothermal Reactor (SHT)

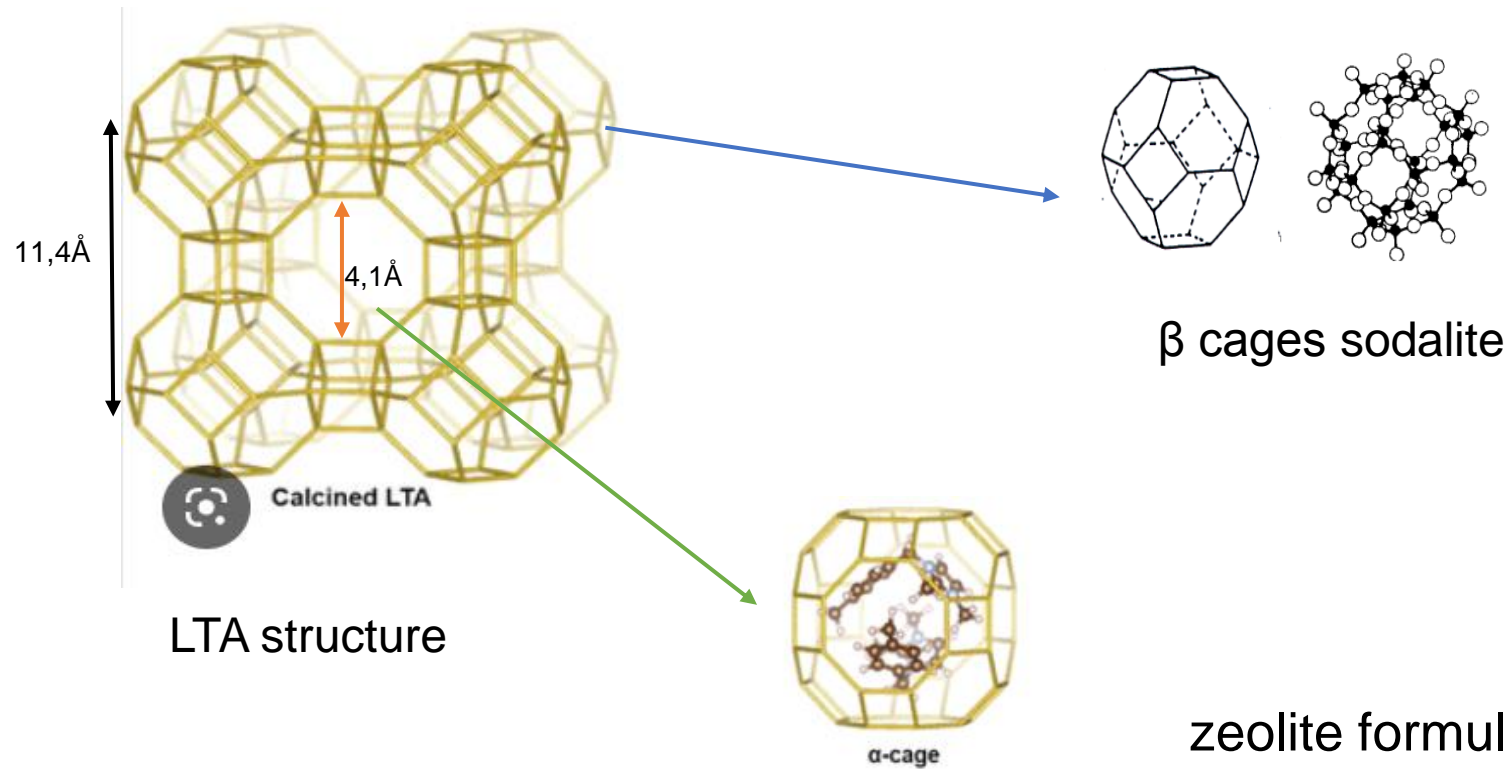


What is Zeolite

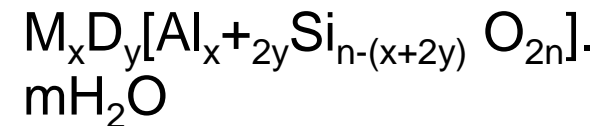


Porous alumino-silica nanomaterial

1756: the Swedish mineralogist **Axel Frederik Cronstedt** the first to introduces zeolite word



zeolite formula :



Eur. J. Mineral. 1998, 10, 1037-1081

The Crystal Chemistry of Zeolithe, E. Paaglia et Richard A. Sheppard

Water waste and
air depollution

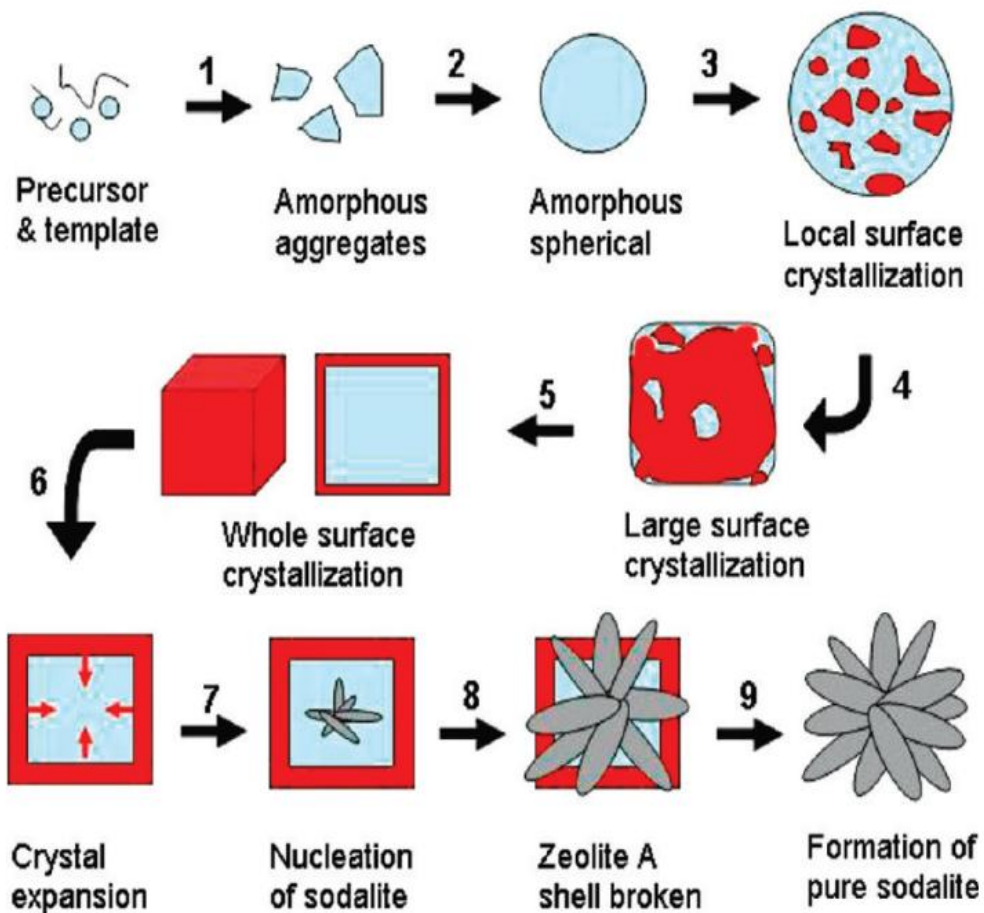
Petrochemistry

Indutrials
application

Heterogeous
catalysis

CO₂ adsorption

NEW ZEOLITE CRYSTALLINE GROWTH PATHWAY AND ZEOLITE PHASE TRANSFORMATION



Greer, et al *J. Am. Chem. Soc.* **131**, 17986–17992 (2009).



Experimental procedure

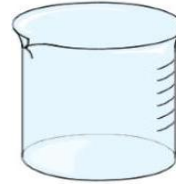


LTA Zeolite Synthesis



**SiO₂ in 3M
sodium hydroxide solution**

+



**NaAlO₂ in
water**

LTA zeolite
formula
 $\text{Na}_{12}(\text{H}_2\text{O})_{278}$
 $\text{Al}_{12}\text{Si}_{12}\text{O}_{48}$

**Mechanical stirring 30 minutes (25 °C)
Or 20 kHz US, 70% amplitude, 30 minutes (70 °C)**



SHT Reactor

SHT experimental conditions:

20 KHz , 100 °C

Autogenous pressure (1,4 atm)

50mL precursor



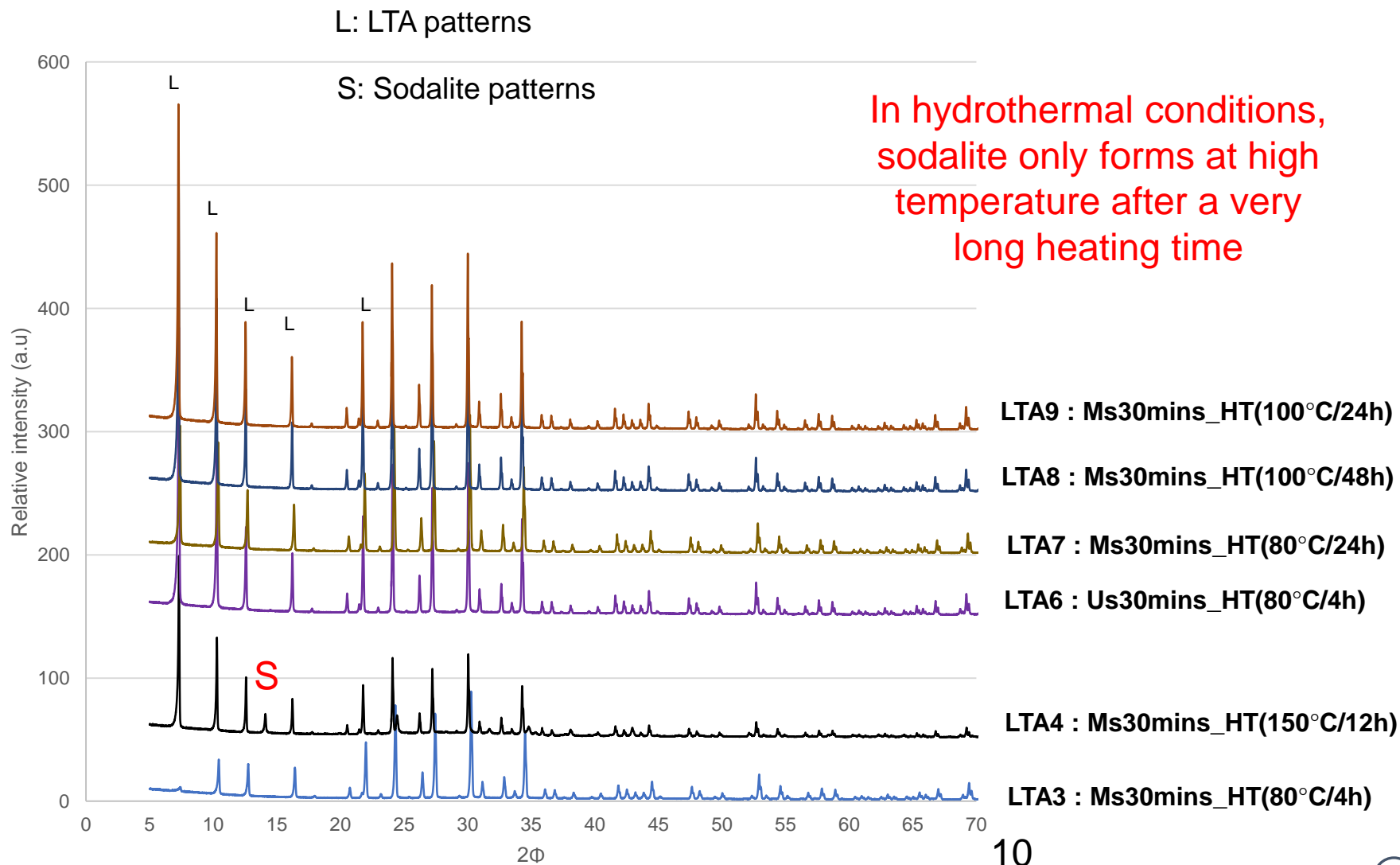
Silent conditions

**Hydrothermal (Parr reactor)
experimental conditions:**

80 °C to 150 °C, 4 h to 12 h



XDR pattern, Hydrothermal synthesis

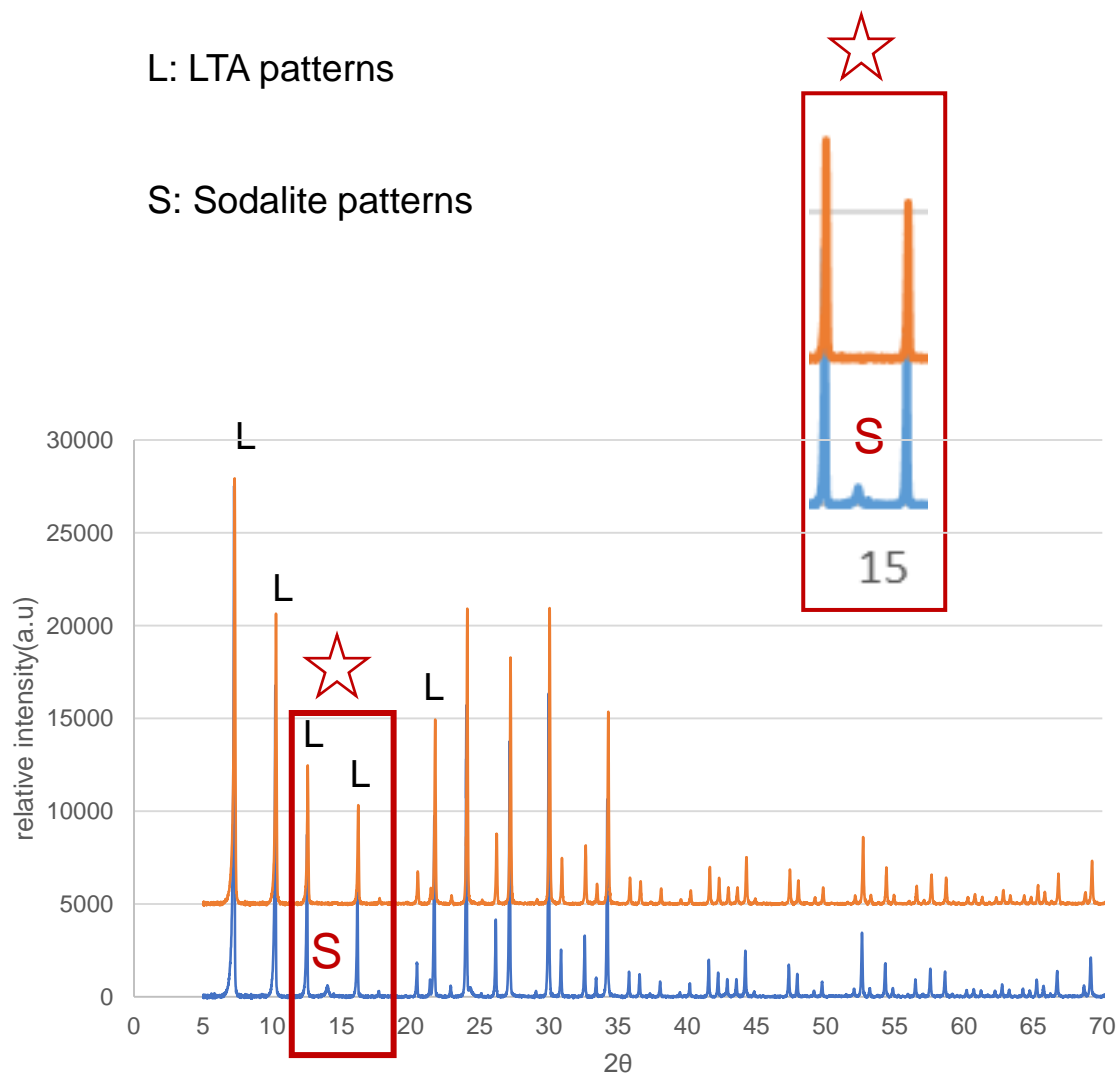


XDR patterns: Sonohydrothermal Synthesis



L: LTA patterns

S: Sodalite patterns

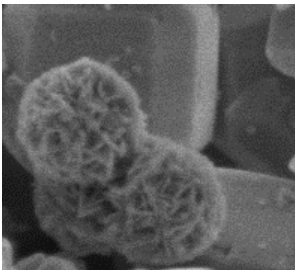
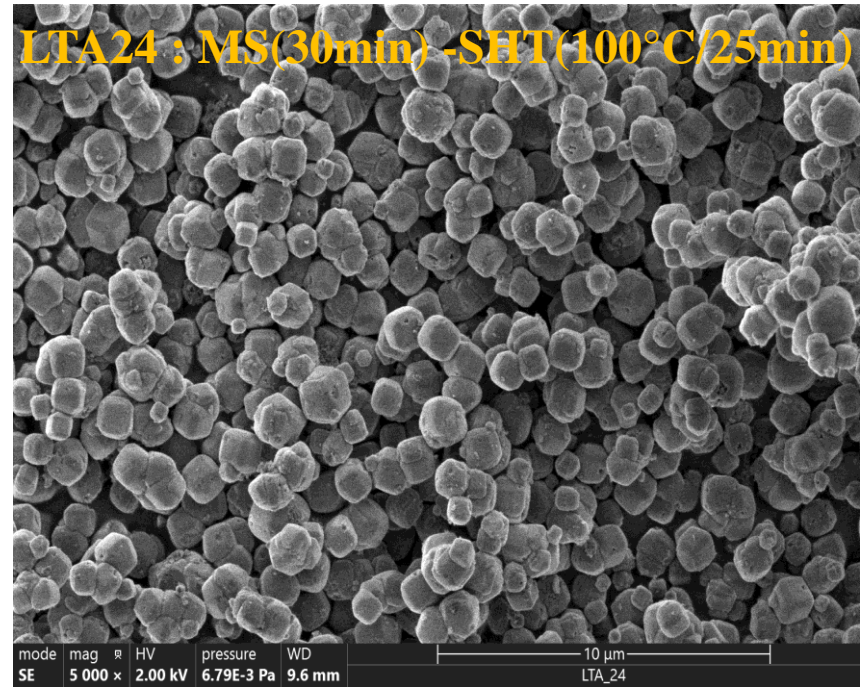
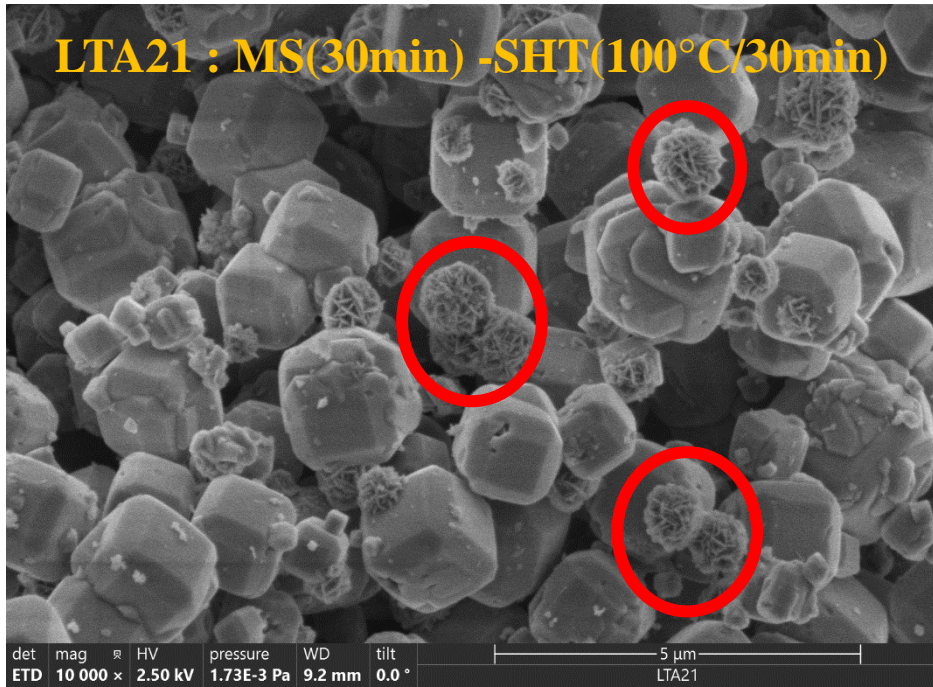


in the SHT reactor,
sodalite is formed at
low temperature in
just 30 minutes

LTA24 : MS(30mins) -SHT(100°C/25mins)

LTA21 : MS(30mins) -SHT(100°C/30mins)

SEM Image of compounds obtain by SHT synthesis



Sodalite morphologie in
rose des sable

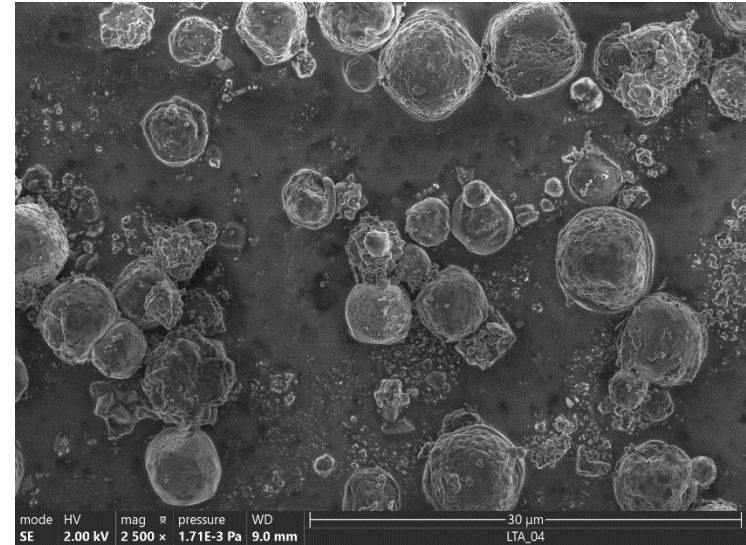
hydrothermal: particule Size (μm)



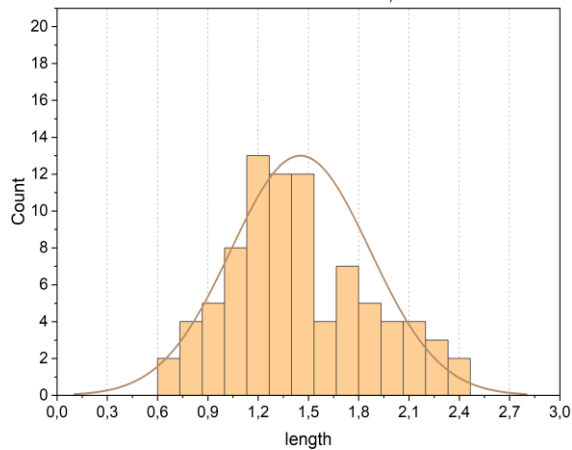
LTA8 : Ms30mins_HT(100°C/48h)



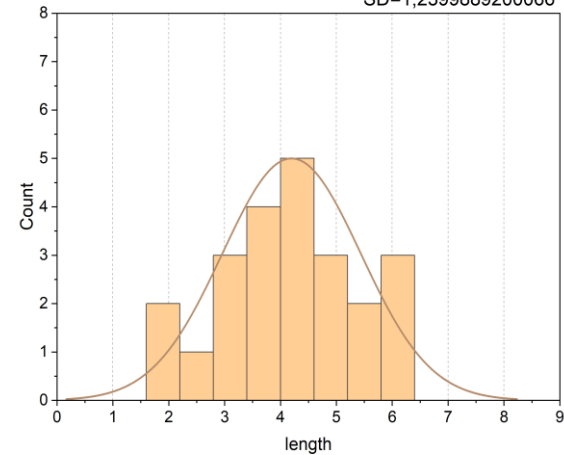
LTA4 : Ms30mins_HT(150°C/12h)



Mean= 1,4524117647059,
SD=0,41474132196546



Mean= 4,1986086956522,
SD=1,2399889200066

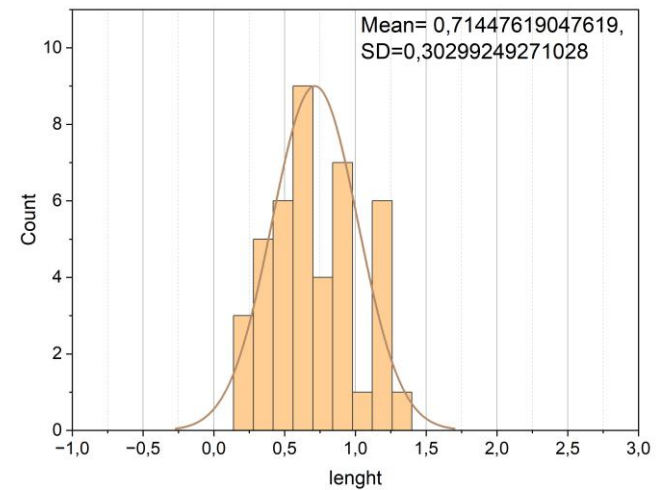
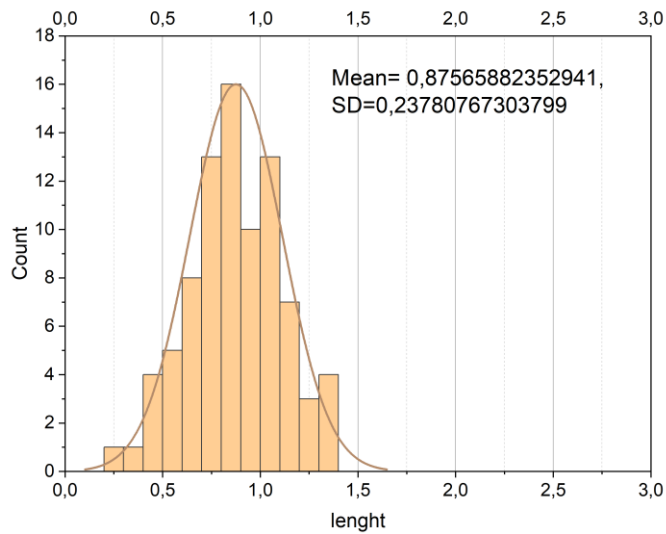
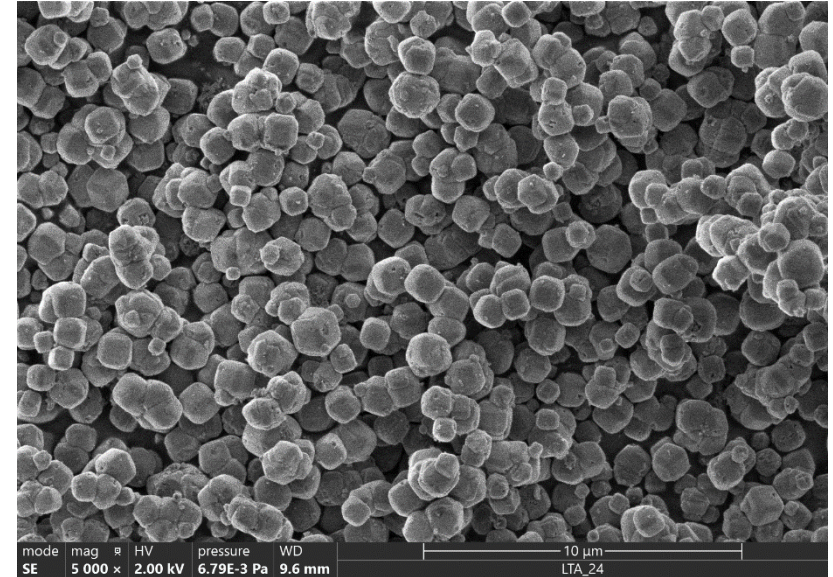
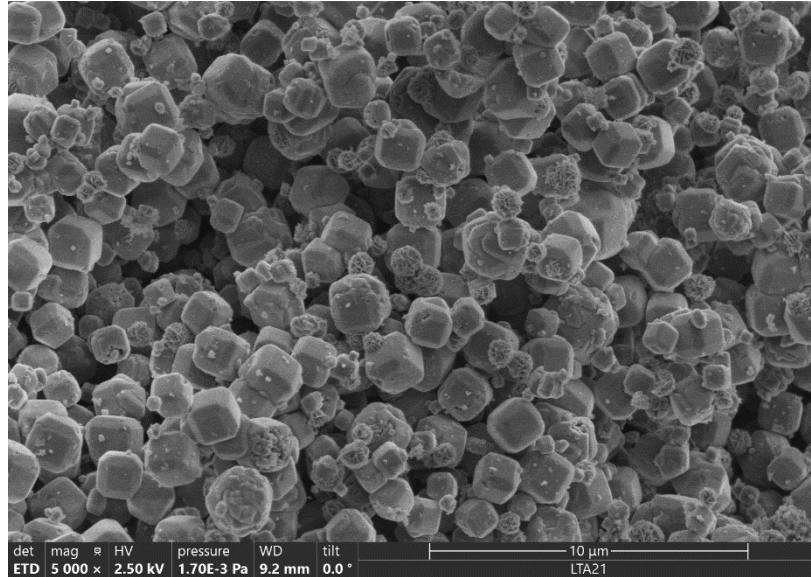


Sonohythermal : particule size (μm)

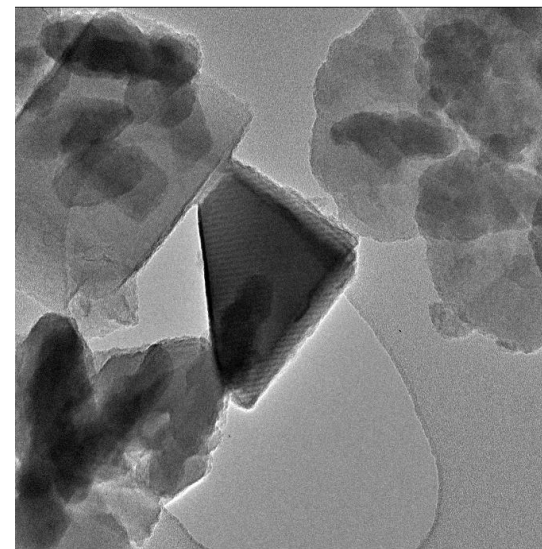
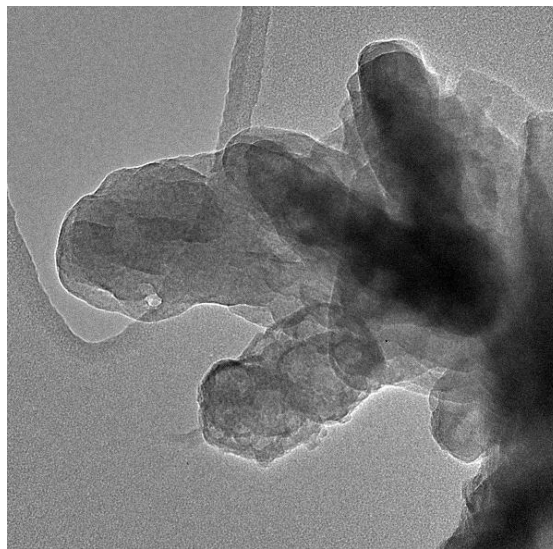
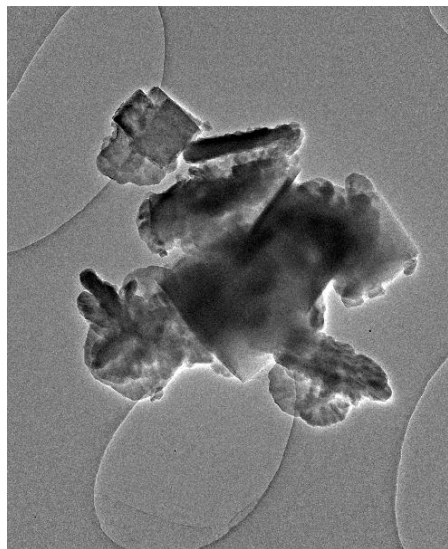


LTA21 : MS(30mins) -SHT(100°C/30mins)

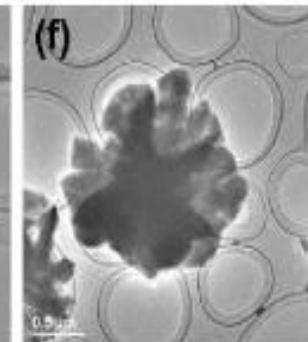
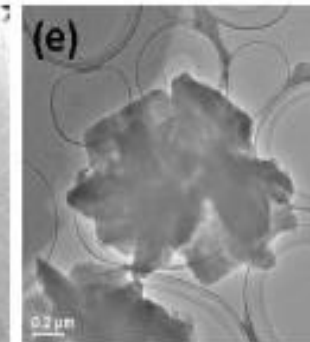
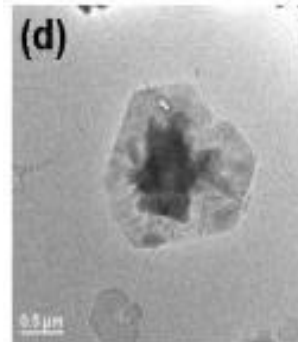
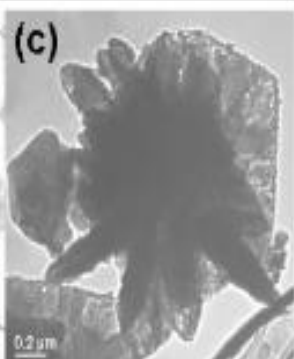
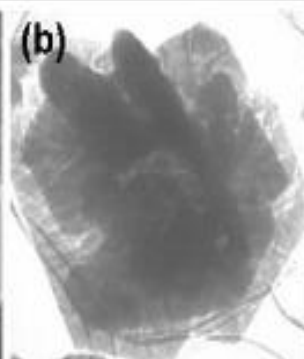
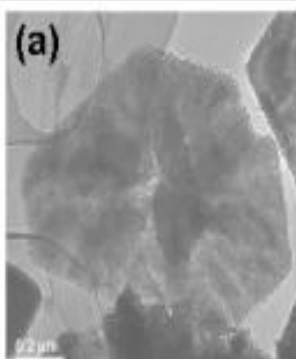
LTA24 : MS(30mins) -SHT(100°C/25mins)



TEM image



LTA21 : MS(30mins) -SHT(100°C/30mins)



Greer, et al *J. Am. Chem. Soc.* **131**, 17986–17992 (2009).

Expected caractérisations

Tomographie

Ultramicrotomie

Fibe

SHT treatment accelerate the kinetic of LTA conversion into sodalite by factor about 48

SHT treatment has an effect on particle size

Perspective

These results pave the way to the synthesis of other zeolites, as well as microporous or hierarchically porous metallosilicates, and maybe MOFs.

thank you for your attention

